A synergy of mechanical loading and oxidation damage in Ni-based superalloys: A critical review

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ABSTRACT
Modern high temperature installations, such as aero-engines and other land-based gas-turbine engines, constitute elevated operation temperatures and lofty pressure ratios, in addition to rotational speeds exceeding 10,000 rpm, necessary for improved engine-efficiencies. Normally, elevated temperatures lead to excessive thermal stresses, while extreme rotational speeds tend to result in tempestuous mechanical stresses, in prohibitively corrosive exhaust gases. Furthermore, high temperatures generally lead to considerable deterioration in material mechanical properties, in addition to a tendency to promoting oxidation damage. Therefore, ideal materials for these applications should exhibit high mechanical-property stability against temperature changes, resistance to oxidation-, fatigue-, and creep-damage. Nickel-based superalloys, materials widely employed for this purpose, are a unique class of materials, designed for manufacture of components that form a backbone of gas turbine-engines. Mechanical behaviour is largely influenced by microstructural features and the synergy between characteristics and constituents of the operating conditions. This paper presents a critical review of the synergetic interaction between mechanical loading and oxidation damage in nickel-based superalloys' behaviour. The review establishes that although oxygen-rich environments were widely cited for the observed enhanced oxidation damage, crack closure effect, reduced creep-load bearing capabilities and accelerated crack growth, to date, only limited work has been conducted to provide a clearer understanding of the mechanism by which interaction between mechanical loading and oxidation damage, lead to inferior mechanical behaviour. Oxidation appears to be a physical process driven by diffusion of oxygen and alloy elements, which tends to be aggravated by the application of a mechanical load. However, the mechanism by which this happens is still yet to be fully understood.

Keywords: Nickel-based superalloys, composition, microstructure, oxidation damage, fatigue loading.

La sinergia entre la carga mecánica y el daño por oxidación en superaleaciones a base de Ni: una revisión crítica

RESUMEN
Las instalaciones modernas con components operando a altas temperatura, como los motores de avión y otros motores de turbinas de gas terrestres, están sometidos a temperaturas de operación elevadas y relaciones de presión elevadas, además de velocidades de rotación superiores a 10.000 rpm, necesarias para mejorar la eficiencia del motor. Normalmente, las temperaturas elevadas conducen a tensiones térmicas excesivas, mientras que las velocidades de rotación extremas tienden a provocar tensiones mecánicas excesivas, en ambientes gaseosos altamente corrosivos como los gases producto de la combustion del motor. Además, las altas temperaturas generalmente provocan un deterioro considerable de las propiedades mecánicas del material, y tendencia a promover el daño por oxidación. Por lo tanto, los materiales ideales para estas aplicaciones deben exhibir una alta estabilidad de las propiedades mecánicas frente a los cambios de temperatura, y una elevada resistencia al daño por oxidación, fatiga y fluencia. Las superaleaciones a base de níquel, son una clase única de materiales, diseñados para la fabricación de componentes que forman la columna vertebral de los motores de turbina de gas. Su comportamiento mecánico está influenciado en gran medida por las características microestructurales y la sinergia entre las características y las condiciones de operación de los components. Este artículo presenta una revisión crítica de la interacción sinérgica entre la carga mecánica y el daño por oxidación en el comportamiento de las superaleaciones a base de níquel. La revisión establece que aunque los ambientes ricos en oxígeno han sido ampliamente citados por incrementar el daño de oxidación, así como el reducir la capacidad de soportar cargas bajo condiciones de creep y el promover el crecimiento acelerado de grietas, hasta la fecha, solo se han realizado un número limitado de investigaciones que proporcionen una comprensión más clara del mecanismo que explique cómo la interacción entre la carga mecánica y el daño por oxidación conduce a una disminución del comportamiento mecánico del sistema.

Palabras claves: Superaleaciones de Níquel, composición, microestructura, daño por oxidación, carga de fatiga.

BACKGROUND

Nickel-based superalloys are materials with excellent mechanical properties at high temperatures. They are designed for the manufacture of components such as turbine blades, guide vanes and discs that form the backbone of the hot sections of gas turbine-engines and other high temperature applications such as nuclear and petrochemical industries. Here, conditions are characterised by high thermal stresses, high mechanical stresses in highly corrosive exhaust gases. Therefore, these environmental conditions, in addition to material microstructure, are known to greatly influence the suitability of a material for an application. Stability of mechanical properties, with operating time, is a critical requirement in structural materials exposed to high static and/or cyclic loads in ambient or non-ambient environments. As such, a material's resistance to degradation due to its reaction with constituents of its environment, notably through the process of oxidation, plays a determining role on the property stability [1, 2]. In modern gas-turbine engines (see figure 1a), high operation temperatures and pressure ratios, in addition to high rotational speeds, are essential as they play a determining role in engine efficiency. However, high temperatures, ranging from 500°C to 1100°C significantly affect mechanical properties [2, 3] and tend to aggravate material oxidation [4]. During operation, turbine blades and discs are subjected to a synergy between oxidation and mechanical loading. On one hand, these conditions lead to fatigue, creep and thermal loading/damage on the material. On the other hand, oxidation brings about material damage due to microstructure change (through composition change) as a result of oxygen diffusion into the material and a counter-diffusion of alloying elements that form the basis of essential microstructural features/phases. As such, material design, manufacture and heat treatment processes normally tend to center on optimization for high strength and damage tolerance. However, chemical complexity resulting from the variety of elements alloyed into nickel, diversity in the manufacturing and heat-treatment processes lead to behavioural specificity [5-9]. This often results from the diversity in the resulting characteristic microstructures, even with the same composition. Due to differences in conditions (levels of temperature and pressure) in the various sections of the turbine-engine (figure 1a), different alloys are considered (figure 1b). Investigation of material behaviour under typical operating conditions is essential in order to ascertain behavioural characteristics.

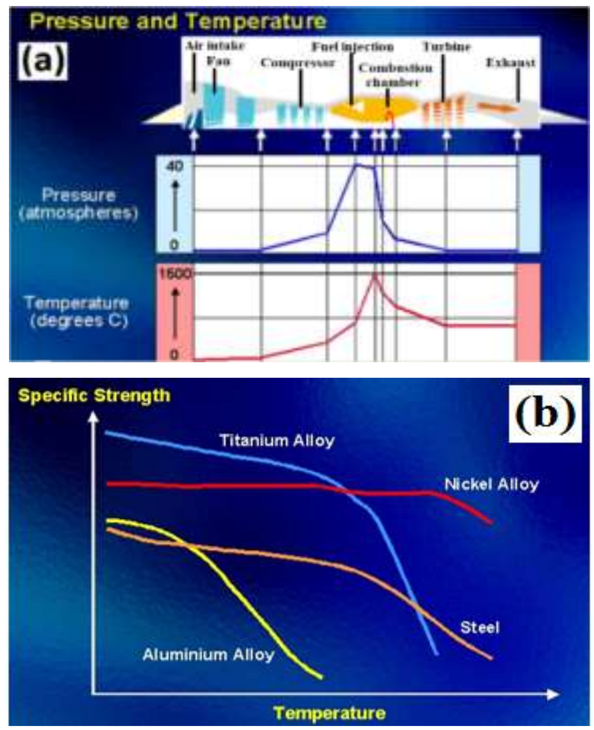

Fig. 1. (a) Variations of temperature and pressure conditions in the turbine, and (b) strength variation, with temperature, for commonly used high-temperature superalloys. (Source: http://www.phase-trans.msm.cam.ac.uk(mphil/Trent1/sld023.thm-12.10.2019).

This [10] is normally aimed at establishing relevant damage mechanisms typically under creep, fatigue and oxidation loading conditions. This paper presents a critical review of the synergetic interaction between mechanical loading and oxidation damage in the behaviour of nickel-based superalloys. Material static or stress-free oxidation behaviour is considered, particularly, effects of the various factors that influence this behaviour are investigated.

Subsequently, after a brief look at the mechanical behaviour, interaction between mechanical loading and oxidation damage is considered. In particular, the influence the synergy, between these two loading conditions, has on the overall low cycle fatigue and crack growth behaviour are investigated.

Static Oxidation behaviour.

Environmental conditions and material microstructure are known to greatly influence suitability of a material for an application. Stability of mechanical properties, with operating time, is a critical requirement in structural materials exposed to high static/cyclic loads in ambient or non-ambient environments. As such, a material's resistance to degradation due to reaction with constituents of its environment, notably through the process of oxidation, plays a determining role on the property stability. This section investigates the influence oxidation has on microstructure stability.

Oxidation in Metals and Alloys.

The process of oxidation is known to be guided and determined by the rate at which reacting species diffuse. As such, most oxidation studies have reported quite rapid rates at the beginning. With time, the process tends to slow down [11-14]. Some studies [15] appear to suggest that the initial rate is rapid due to the fact that the reacting species are readily in contact at the beginning of the process. Subsequently, diffusion determines the rate at which the process will proceed. Diffusion of reacting species (oxygen and metal atoms) through the formed oxides is observed to be much slower than through material bulks. Others have suggested that due to the differences in atomic arrangement between the material bulk and the formed oxides, atomic diffusion rates through them, for the reacting species, will also be different. Lower diffusivities have, therefore, been associated with oxides as opposed to material bulks [12]. Oxidation rates, therefore, tend to reduce significantly as the thickness of the oxide grows.

Different characteristics of oxidation were reported. Some metals were found to form only one type of oxides while others formed different types depending on prevailing conditions. Metals such as Al, Zn and Ni formed only one type of oxide, irrespective of the oxidation conditions [12, 16]. However, although different crystal structures of Al oxides were observed, no distinctly different crystal structures were observed in oxides of Zn and Ni. On the other hand, Fe, Cu and Co appeared to oxidise in a manner different from Al, Zn and Ni. Here, two factors were found to influence the process. These were the level of temperature and stoichiometry (amount of oxygen available for oxidation). It was noted that a lean oxidation led to the formation of unstable oxides of the form FeO and CoO. Rich oxidation, however, were reported to lead to the formation of the more stable oxides Fe_xO_y and Co_xO_y, where x and y are integers greater than 1 [17-19]. Due to a limited availability of oxygen at the oxide-metal interface as compared to the oxide-environment interface, multiple layers of oxides of the form FeO and CoO formed next to the metal. On the other hand, although Cr oxidises in a similar manner as Fe, Cu and Co, one of the forms of the oxide has been found to be volatile under certain conditions. At higher temperatures, oxides of the form CrO_3 easily evaporated. As such, certain engineering materials that rely on formation of chromia (chromium oxide) as protection against further oxidation, are known to have a temperature limitation [12, 20, 21].

Oxidation Damage in Ni-based Superalloys.

At temperatures in excess of 500°C, oxidation is known to contribute considerably to a deterioration in material mechanical properties, particularly, fatigue/creep capabilities. Based on the composition-microstructure-property paradigm, formation of surface and internal oxides changes composition, leading to microstructure change which ultimately changes mechanical properties. This leads to increased formation of cracks due to the brittle nature of the formed oxides which tend to spall off at increased rates due to the presence of a mechanical load. The oxidation process constitute a combination of oxygen diffusion into the material and a counter-diffusion of alloying elements towards the surface [22-24].

Oxidation behaviour, as reported in literature [25-27], is generally characterised in terms of oxidation kinetics as well as the change in material composition with respect to both time and temperature. This is in line with the work of Giggins and Pettit [28, 29]. However, formation of certain oxides, such as alumina and chromia, is known to curtail further oxidation in some superalloys. As such, oxidation of nickel-based superalloys containing significant amounts of Al and Cr results in this phenomenon [27]. Oxidation rates of most nickel-based superalloys had a tendency of reducing with time. Literature [27, 30], therefore, recognises that oxidation kinetics are generally assumed to conform to linear, parabolic or logarithmic laws where mass changes are normalised on the basis of specimen surface area.

The major oxidation damage mechanism refers to the formation of surface and internal oxides. These oxides are of elements that form the basis for critical phases, the *γ-matrix* and *γ'-precipitates*. Diffusion of alloying elements from these phases towards the external surface resulted in microstructural change due to the denudation of strengthening phases (the *γ'-precipitates*). Other forms of damage reported included void formation and spallation. Furthermore, most recent studies have reported the formation of multi-layered oxide scales. An outer layer comprised mainly of NiO and Cr_2O_3 as well as traces of CoO in some cases. Various and complex oxides, commonly referred to as spinels, laid beneath the outer layer, including $NiTiO_3$, $NiTaO_4$ and $(Ni/Co)Cr_2O_4$. The third layer was composed mainly of discontinuous oxides, predominantly Al_2O_3, as shown in figure 2a and b [31, 32]. Various depths of regions, deficient in the strengthening phases, the *γ'-precipitates*, were also reported directly beneath the third oxide layers [31, 33, 34]**.**

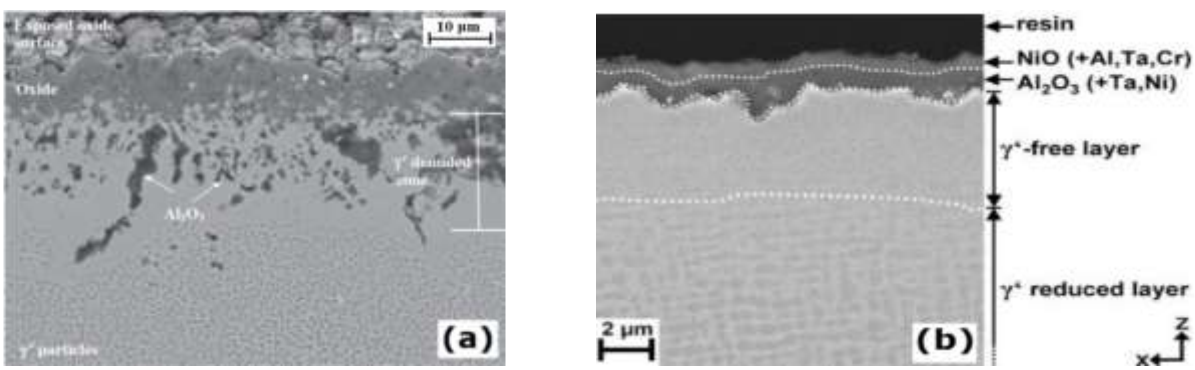

Fig. 2. Nature of internal and external oxides, and *γ'-precipitate* free region in (a) a PM alloy RR1000 oxidised at 800ºC [4], and (b) a single crystal alloy oxidised at 980ºC [31].

Similar results were also reported in directionally solidified alloys [35]. Additionally, measurable weight losses in test specimens were reported in some studies. These were linked to the formation of void and spallation of the formed oxides [36]. One recent study appeared to suggest that spallation was mostly a result of thermal cycling [37]. Others [38-40] were of the view that the presence of sulfur (S) in the alloy promoted formation of voids, cracks and pores along oxide/metal interfaces, leading to increased spallation. Generally, oxidation alters material composition which subsequently leads to a change in microstructure and thus material property deterioration. However, although oxidation kinetics are generally assumed to conform to either linear, parabolic or logarithmic laws, kinetics for individual oxides (NiO, Cr_2O_3, Al_2O_3 and spinels – $NiTiO_3$, $NiTaO_4$ and $(Ni/Co)Cr_2O_4$) are still not yet fully understood.

Effects of Microstructure.

Microstructural features play crucial roles in the way nickel-based superalloys resist oxidation damage. Phases such as carbides, borides and other secondary phases, in addition to the *γ-matrix*, *γ'-precipitates* and grain-structure, tend to influence oxidation behaviour to a large extent. A study by Zhao *et al.* [21] has demonstrated that reduction in the size of *γ'-precipitates* can lead to improved resistance to oxidation. Two alloys with almost similar chemical composition, but different *γ'-precipitate* sizes, were studied. The alloy with finer *γ'-particles* exhibited high resistance to oxidation as compared to one with coarser ones.

In the alloy with a finer *γ'-precipitate* structure, this was a result of an increased number of possible nucleation sites for oxides that had a tendency to retard oxidation, particularly, aluminum oxide (Al_2O_3). It was suggested that a larger surface area of *γ'-precipitates* was exposed to oxygen. This was said to lead to a more rapid formation of Al_2O_3. Additionally, the study was of the view that, the alloy with a finer *γ'-precipitate* structure presented an increased phase boundary which appeared to have a higher Al diffusivity. This led to enhanced outward diffusion of Al which formed a protective oxide (Al_2O_3). Other studies, such as those by Wang *et al.* [41] reported similar findings. On the other hand, although Mallikarjuna *et al.* [42] appear to suggest otherwise, their results were in agreement with the findings reported by Wang *et al.* [41] and Zhao *et al.* [21], in terms of the formation of the *γ'-free zone*.

Considering oxidation behaviour of alloys with different grain-structures, studies have shown that the presence or absence of grain boundaries considerably affects the oxidation behaviour. In materials produced through the PM route (traditional polycrystalline materials), grain boundary strengthener phases, such borides and carbides and other secondary phases, were formed from the presence of B, C, Hf and Zr. Since carbon is known to have a high affinity for elements such as Hf, Zr, Ta, Ti Nb, W, Mo, V and most notably Cr, carbides of these elements are formed at solidification stage. In a study that subjected a traditional polycrystalline nickel-based superalloy to fatigue and high temperature, oxides rich in Cr and Co were reported along grain boundaries [43].

However, some studies [4] reported discontinuous oxides of Al along grain boundaries (see figure 3a). Grain boundary oxidation was also characterized by void formation. As shown in figure 3 [26], the amount of voids tends to increase significantly with the increase of temperature. This, however, is not the case for single crystal alloys. Even at much higher temperatures, oxidation in single crystal alloys appear to cause limited internal damage when compared to polycrystalline alloys, although both types of alloys tend to form γ'-free regions beneath oxide layers.

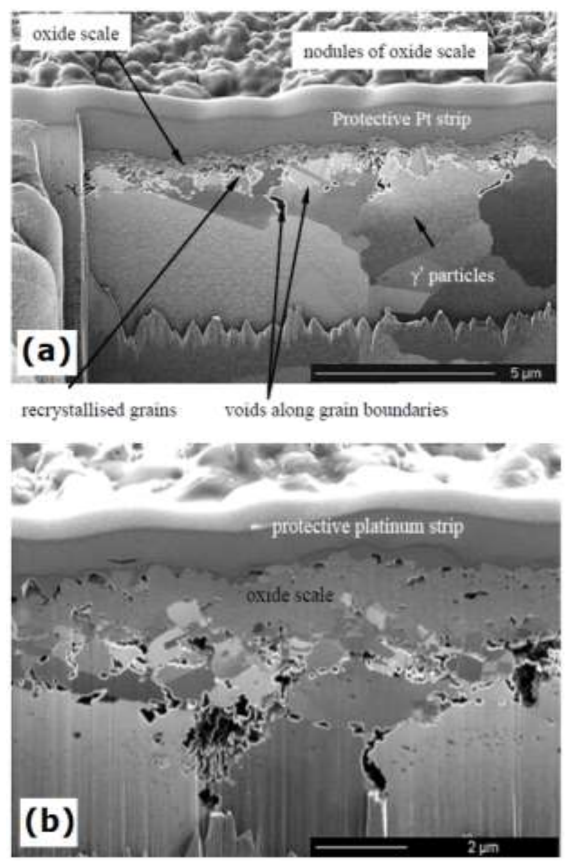

Fig. 3. Damage in a powder metallurgy alloy RR1000 oxidised at (a) 700ºC, and (b) 800ºC [26].

The different oxidation behaviour between single crystals and polycrystals can be seen from figure 2. The high resistance to oxidation damage in single crystals could be one of the reasons why they are preferably used as turbine blade materials, where conditions are harsher. Polycrystals on the other hand are used as turbine discs working in less harsh conditions. Studies [44, 45] investigating grain size influences on the oxidation behaviour of polycrystalline superalloys reported higher resistance to oxidation in fine-grained materials than in coarse-grained materials. The fine-grained alloys had a greater grain boundary surface area which allowed more oxygen diffusion into the bulk. This was not the case in the coarse-grained alloys, as the reduced grain boundary surface area only allowed limited oxygen diffusion into the bulk. The increased oxygen diffusion into the fine-grained alloys led to the formation of more internal alumina/chromia protective oxide layers. This inhibited further oxidation resulting in reduced oxidation rates (observed in the kinetics), as compared to the coarse-grained alloys. This is what led to the difference in oxidation between the fine-grained and coarse-grained alloys. It is noteworthy, however, that the synergetic influence of phase and grain structure was not looked into. Literature is still deficient in knowledge about the optimum sizes of the *γ'-precipitates* and grains are necessary for a polycrystalline material to be oxidation damage resistant. Furthermore, to the best of our knowledge, no study has attempted to established which of the two microstructural features (*γ'-precipitates* or grain boundaries) accounts for higher oxidation damage in a polycrystalline material.

Effects of Temperature.

Temperature is a critical parameter in the operation of modern gas-turbines as it directly determines the overall efficiency of the system. Investigations into the tensile deformation behaviour, at various temperatures, of nickel-based superalloys revealed that mechanical properties were temperature dependent [46]. This was also true for the deformation behaviour of individual phases (*γ-matrix* and *γ'-precipitates*). Here, deformation behaviour, such as modulus of elasticity, for the two phases reduced with an increase in testing temperature [3].

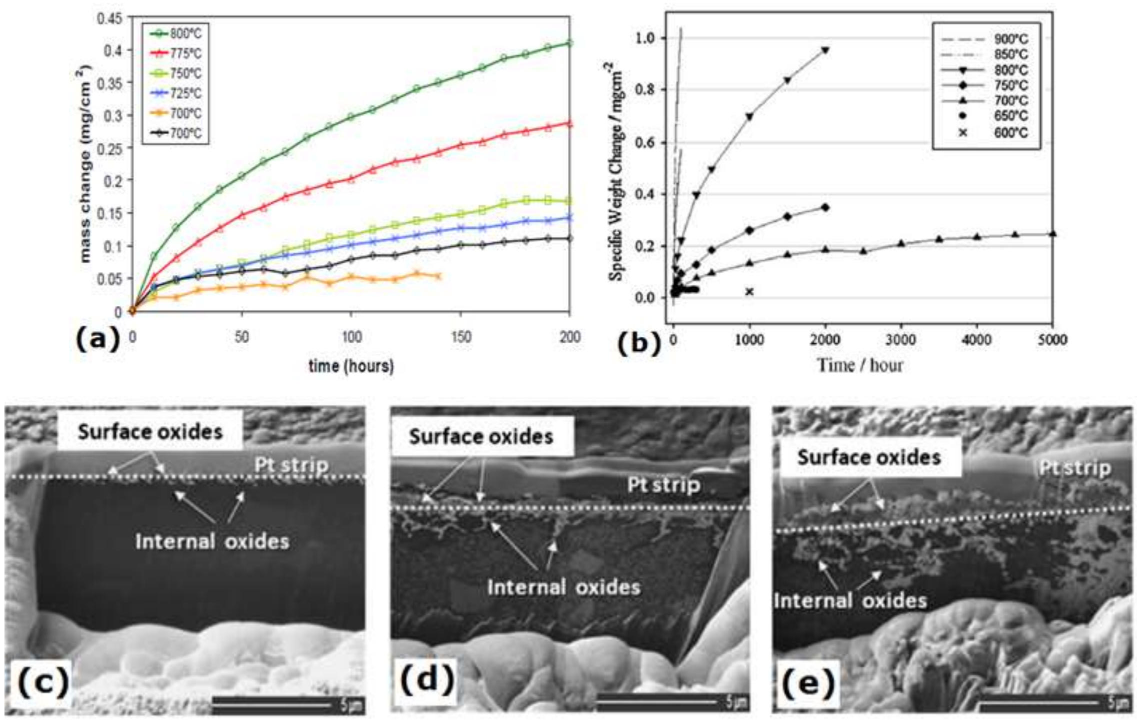

Fig. 4. Temperature-dependency of oxidation of nickel-based superalloys. Kinetics of (a) a polycrystalline alloy RR1000 [26], and (b) single crystal Rene N5 at various temperatures [4]. Cross-sectional analysis of oxidized polycrystalline alloy RR1000 at (c) 700ºC, (d) 750ºC, and (e) 800ºC [47].

Oxidation behaviour of nickel-based superalloys is highly dependent on temperature, as reported in a polycrystalline material at temperatures in the range of 600-900°C [48]. It was observed that rates of oxidation drastically increased as temperatures increased. Elsewhere, this was reported in both polycrystalline and single crystal nickel-based superalloys, as evident from the oxidation kinetics at different temperatures shown in figure 4a and 4b [4, 26]. This entails that as temperature increases, a material's resistance to oxidation reduces, making it more susceptible to failure if a mechanical load is applied.

Karabela *et al.*, [46] investigated the effect of temperature on the oxidation behaviour of a powder metallurgy nickel-based superalloy (RR1000). Three temperatures, 700ºC, 750ºC and 800ºC, were considered. Focused-ion beam (FIB) sectioning and secondary ion imaging were used to analyze the extent and depth of damage at the respective temperature. Results showed significant differences in the extent of damage at different temperatures, shown in figure 4c, 4d and 4e. It was confirmed that high temperature leads to increased material damage. Under similar conditions, oxidation at 800ºC resulted in internal oxides being far much deeper and widespread than at 700ºC. Similar results were reported elsewhere as shown in figure 3 [26, 49-51].

Interaction of Mechanical Loading and Oxidation Damage.

During operation, turbine blades and discs are subjected to a synergy between oxidation and mechanical loading. On one hand, these conditions lead to fatigue, creep and thermal loading on the material. On the other hand, oxidation brings about material damage due to microstructure change as a result of the diffusion of oxygen into the material and a counter-diffusion of alloying elements that form the basis of essential microstructural features/phases. As such, material design, manufacture and heat treatment processes normally center on optimization for high strength and damage tolerance. Investigation of their behaviour under typical operating conditions, therefore, is essential in order to ascertain behavioural characteristics. This is normally aimed at establishing relevant damage mechanisms typically under creep, fatigue and oxidation loading conditions [10]. In this section, interaction between mechanical loading and oxidation at high-temperature in nickel-based superalloys is reviewed. The following covers the effects of oxidation on mechanical behaviour, effects of mechanical loading on oxidation behaviour and recent developments in the modelling of the interaction between oxidation and mechanical loading.

Effects of Oxidation on Mechanical behaviour.

Under an applied mechanical load at high temperature and in oxygen-rich environments, one of the major causes of inferior mechanical behaviour is thought to be dynamic embrittlement [52-54]. Here, increased material cracking was found to be driven by the presence of oxygen. A study by Molins *et al.* [55] suggested that embrittlement is caused by a combination of oxidation and mechanical loading at and ahead of a crack tip. Furthermore, the study suggested that the formed oxides would consist of layers with the same structure as those reported for smooth specimens tested under static conditions. However, attempts to provide evidence for this phenomenon were only presented recently by Kitaguchi *et al.* [47]. Three cases were considered, i.e., a growing crack as well as a stationary crack with and without a mechanical load. Results showed that although the two stationary cracks showed slightly more oxidation than the growing one, similar oxidation mechanisms (in terms of layers of formed oxides) were observed for all the three crack tips. In another study by Viskari *et al.* [57], a polycrystalline (Allvac 718Plus) nickel-based superalloy was subjected to a constant load for a duration of 600 s, and crack tip cross-sections were analysed. In line with the suggestion by Molins *et al.* [55] and also similar to the findings reported in stress-free oxidation studies in literature [31, 33, 58], multi-layered oxide structures were formed and found at the crack tip as well as immediately ahead of it. In this case, Viskari *et al.* [57] envisaged embrittling oxygen atoms to have diffused through grain boundaries ahead of the fully opened crack. The study suggested that formation of oxides ahead of a crack tip leads to increased material cracking and thereby bringing about the inferior mechanical behaviour, widely reported in oxygen-rich environments. In other studies [52-54] investigating effects of oxidation on mechanical behaviour, no evidence of oxides at the crack tip was reported. Oxides were only found on crack flanks behind the crack tip. Further investigations showed that increased grain boundary decohesion was only dependent on oxygen partial pressure [52-54]. Other studies such as those by He *et al.* [59], investigated the effect of oxidation on the crack growth behaviour, noted that fatigue crack propagation was temperature-dependent. It was further observed that crack propagation rates at higher temperatures had a tendency to slow down. At lower temperatures, this was not the case, as crack growth rates appeared to speed up more rapidly and even exceeded those at higher temperatures. Crack closure effect, resulting from the oxidation of crack flanks and leading to a reduction in the effective load ($P_{max} - P_{min} = \Delta P$), was cited as the cause for the observed behaviour [59]. Similar findings were reported elsewhere in literature [14, 60–63]. Generally, formation of oxides results in the weakening of the material (near the surface) through the change in the underlying microstructure, resulting in inferior mechanical load-bearing capabilities. Studies on creep behaviour of nickel-based superalloys reported significant reductions in creep-life in air as compared to vacuum conditions where oxidation is immeasurably low [31, 33]. Other studies [64-66] only appeared to suggest that prolonged material exposure to high temperature oxidation led to deterioration of mechanical properties.

Effects of Mechanical Loading on Oxidation behaviour.

Some of the early studies on the influence mechanical loadings have on the oxidation behaviour were published in the late 1990's by a group of researchers who considered various loading regimes [67]. Loading regimes considered included cyclic, creep and a combination of the two. In the study, oxidation was considered as the diffusion of oxygen into the material, and an investigation into the different diffusion rates, at specified temperatures and oxygen partial pressures, in the material bulk and in the formed oxides were conducted. While creep testing was conducted with a tensile load less than the elastic limit and fully-reversed fatigue testing was done with a strain range of 0.1%, fatigue-creep testing was carried out by imposing a 15 mins dwell in the fatigue loading cycle. Results showed higher initial rates of oxidation in all the considered loading regimes. Subsequent rates of oxidation were somewhat reduced and appeared more stabilised. This was in line with the findings obtainable in literature [15, 68], where three stages of oxidation were identified for oxidation kinetics. The initial stage appeared almost linear, rapid and quite short. It was suggested that this was a result of the fact that reactants were readily in contact at the beginning of the oxidation process. Thereafter, the rate of diffusion of the reacting species, through the formed oxide, determined the oxidation rate. Generally, the study concluded that the presence of mechanical loadings (be it fatigue, creep or fatigue-creep) resulted in enhanced oxygen diffusion [67]. Other studies have investigated the effect of a static tensile and compressive mechanical loading on the oxidation kinetics. Material chemical composition had traces of C, Si, S, Fe and Co with nickel accounting for 99.9853%. As the load level increased, enhanced oxidation behaviour was observed. This culminated into increased mass gains with time as shown in figure 5b, irrespective of the type of loading; tensile or compressive [69, 70]. Similar results were reported in a study that investigated the effect of fatigue loading on the oxidation kinetics of Cr-Mo steel [71]. It is noteworthy, however, that Moulin *et al.* [67] and others [69, 70] considered the oxidation behaviour of pure nickel, which is of less practical application in the current state of the art. In alloys (where up to more than 10 elements are added, such as Ni-based superalloys), the oxidation behaviour becomes more complex than in pure metals. Additionally, the study only considered the diffusion of oxygen into the material and neglected the counter-diffusion of the material element(s). To date, influence of mechanical loading on oxidation kinetics has not been fully explored in nickel-based superalloys.

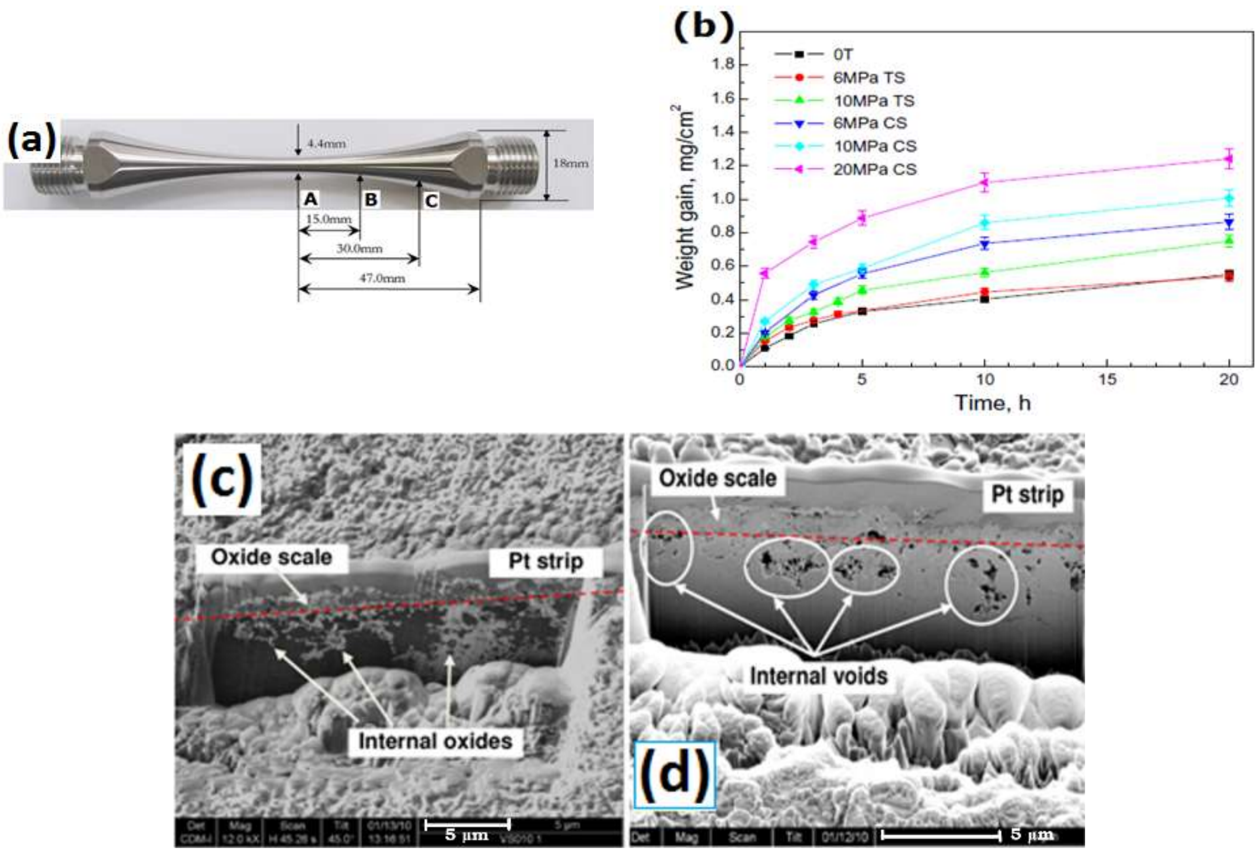

Fig. 5. (a) Specimen used to study the influence of stress level (at *A*, *B* and *C*) on the oxidation behaviour [40]; (b) Load-dependency for the kinetics of oxidation for pure nickel, at 700ºC [69]; Cross-sectional analysis of oxidation at location (c) *A* and (d) *B* [40].

In a recent study, the influence of a cyclic load on the oxidation behaviour of a nickel alloy was investigated at various temperatures. The material studied was a nickel-based superalloy produced through powder metallurgy route, with 10 elements alloyed into it. This produce a traditional polycrystalline alloy with uniform grain size range of 4 - 5 µm. To provide variable stress levels, waisted specimens were machined (see figure 5a), with diameter changing from 4.4 mm in the middle (location *A*) to a maximum of 18 mm at the ends (near location *C*) and an intermediate diameter at location *B*. Load-control fatigue testing was carried out at load-level of 10 kN at temperatures of 700°C and 750°C. At 800°C (due to reduced properties) a reduced load level of 8 kN was considered. A loading frequency of $f = 0.25$ Hz at a load ratio of $R_L = 0.1$ [40]. Influence of stress level on oxidation behaviour was then studied by measuring oxidation damage at the three locations *A*, *B* and *C* (figure 5a).

Cyclic tests were conducted till fracture which occurred after durations of 196 h (700ºC), 194 h (750ºC) and 34 h (800ºC). Results showed that oxidation damage was considerably high and stress-dependent. At a temperature of 800ºC, location *A* in figure 5a exhibited the deepest penetration of oxygen, evident from the micrographs in figure 5c. Reduced depth of penetration was observed at location *B* (figure 5d) as compared to location *A*. Much reduced oxidation was observed at location *C*. It was concluded that the level of stress had a significant effect on the oxidation behaviour of nickel-based superalloys. However, the study was not able to comment on the kinetics of the oxidation at the various locations (with different stress levels).

Generally, in both metals and alloys, mechanical loading (irrespective of creep, fatigue or a combination) appears to lead to enhanced extents of oxidation damage. This could be a result of increased oxygen ingress into the material due to the stretching of lattice sites arising from the presence of a mechanical load.

Synergy of Mechanical Loading and Oxidation Damage.

Experimental studies have widely demonstrated and reported the synergetic interaction between mechanical loading and oxidation damage. As compared to vacuum conditions, this mostly resulted in enhanced oxidation damage [40], crack closure effect [14], reduced creep load bearing capabilities [31] and accelerated crack growth behaviour [72]. However, to date, only limited studies were conducted on the modelling of the interaction between mechanical loading and oxidation, in nickel-based superalloys. Of interest to this study are the findings of a computational-based study that investigated the influence of a mechanical load on the oxygen diffusion into a nickel-based superalloy along grain boundaries.

Simulations of oxygen diffusion were controlled by the parabolic oxidation rate and diffusivity and carried out at temperatures in the range of 750°C to 800°C. To study the influence of mechanical loading, natural diffusion and coupled deformation-diffusion simulations were conducted. One-way coupled deformation-diffusion simulations were carried out with stress-assisted oxygen diffusion driven by the gradient of the hydrostatic stress. Simulation results for natural diffusion and stress-assisted diffusion at 650°C revealed enhanced depths of oxygen diffusion/penetration. The rate of change of oxygen concentration at the crack tip during stress-assisted diffusion was far higher than that for natural diffusion, as shown in figure 6a. These results are in line with the experimental findings (kinetics) of Zhou *et al.* [69] who studied the oxidation of pure nickel (figure 6b). A comparison of the depth of penetration of oxygen due to natural diffusion (figure 6b) and stress-assisted diffusion (figure 6c) reveals significantly increased oxidation damage [74].

Nevertheless, although the study was able to successfully model oxygen diffusion as observed from experimental studies, in the numerical computation, diffusion was restricted to grain boundaries only. This contradicts with literature [26, 75] where both grain boundaries and bulk oxidation was reported. Further studies have simulated oxidation damage without restricting oxygen diffusion to grain boundaries.

The studies also considered the effects of dwell imposed at peak load on the depth of oxygen penetration. It is clear that imposition of a dwell at peak load leads to increased diffusion of oxygen into the crack tip, as shown in figure 6f. An increase in oxygen penetration and concentration was observed under fatigue (figure 6e) when compared to natural diffusion (figure 6d). Although these studies were able to reflect the physical effects of mechanical loading and oxygen diffusion interaction as envisaged by Andrieu *et al.* [76] and Moulin *et al.* [67], there was a lack of validation against test results. Furthermore, critical diffusion parameters such as the oxidation parabolic rate constant, diffusivity and pressure factor, were only estimated from literature.

Karabela *et al.* [47] has modelled the effects of a cyclic load on the oxidation of a polycrystalline superalloy, by employing an approach similar to those applied in Zhao [74] and Zhao *et al.* [77]. Results were validated by the findings of an experimental study by Karabela *et al.* [40], as shown in figure 5a, 5c and 5d. Here, although the oxidation parabolic rate constant was estimated from literature, diffusivity and pressure factor were measured based on test results.

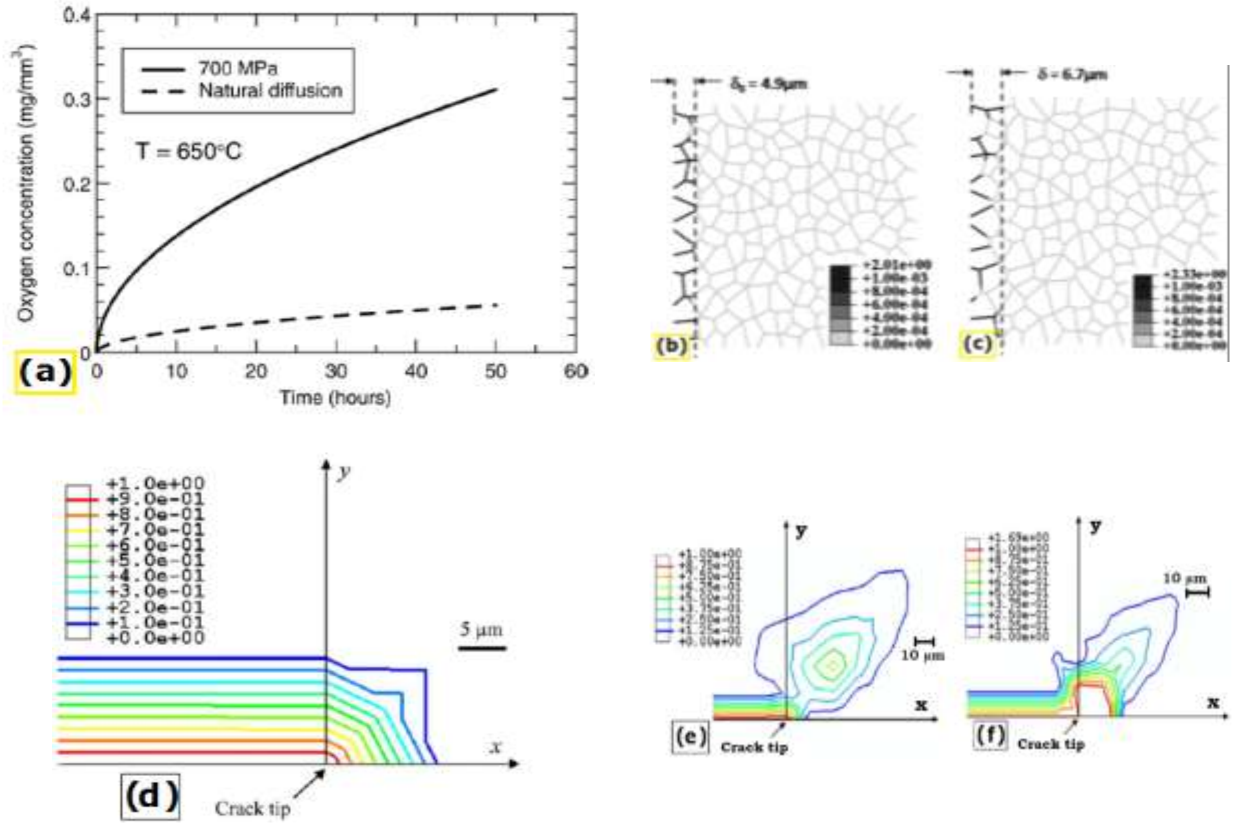

Fig. 6. (a) Natural and stress-assisted oxidation kinetics at 650°C. Contour plots for (b) natural diffusion of oxygen along grain boundaries, and (c) stress-assisted diffusion along grain boundaries (under a 1100 MPa creep load) [74]. Contour plots for (d) natural diffusion of oxygen (e) fatigue-assisted diffusion of oxygen and (f) fatigue-assisted oxygen diffusion with 10 s dwell [77].

The pressure factor was calibrated based on the depth of oxygen penetration. This was achieved by varying the pressure factor values and running FE simulations for the duration of the test, till the depth of oxygen penetration, observed in tests, was achieved. To obtain pressure factor values at other temperatures, the process was repeated. The obtained values of pressure factor were then utilized in carrying out FE simulations for predicting crack growth under fatigue-oxidation conditions. Based on the modelled interaction between mechanical loading and oxidation in Zhao *et al.* [46] and Karabela *et al.* [73], crack tip deformation behaviour under fatigue-oxidation conditions was studied. Effects of mechanical loading on oxygen diffusion was considered but effects of oxygen diffusion on deformation were completely neglected. Particularly, the studies only considered stress-assisted diffusion whilst ignoring the compressive stress generated by dilatation strain resulting from oxygen penetration into the material. Literature, such as Wu [22], Suo *et al.* [23] and Elkadiri *et al.* [24], have outlined that oxidation by itself induces a compressive stress into the material. By only considering the effect of stress on diffusion, Zhao *et al.* [46] and Karabela *et al.* [73] neglected the influence of the oxidation-generated material-expansion on the stress state. Several experimental studies [31, 41-43, 75, 76, 78] have cited oxidation as a cause for reduced mechanical behaviour in nickel-based superalloys. Studies such as Evangelou *et al.* [79] reported a myriad of competing factors, in addition to the effects of oxidation. This was partly due to the temperature level (550°C) at which tests were conducted. In a study on thermomechanical fatigue behaviour of a single crystalline nickel-based superalloy [80], a fatigue-oxidation damage was reported to dominate material behaviour in the out-of-phase loading mode, while damage in the in-phase loading mode was mainly a result of creep-fatigue. Others [81], reported extensive crack flank oxidation and were of the view that damage was a result of the interaction between creep, fatigue and oxidation. As such, prediction of life for components subjected to mechanical loadings in oxygen-rich environments is an area requiring concerted research attention. He *et al*. [59] has presented a model to predict fatigue crack growth in a DS nickel-based superalloy. The model was based on modifying the Paris law, by making the constant *C* and exponent *m* temperature-dependent and incorporating a thermal activation energy parameter to account for the effects of oxidation. To predict the different fatigue crack growth rates observed in the different loading directions (with respect to the orientation of columnar grains), different values of the activation energy were proposed. However, according to the findings in other studies [33, 34, 37, 55, 56, 82], changing of material orientation has little effect on the oxidation behaviour. Nevertheless, it is recognised that deformation is strongly influenced by crystallographic orientation [83-85]. Therefore, since deformation greatly influences the extent of oxidation [40], the different values of the activation energy reported in the considered orientations could have resulted from the different deformation behaviour. Other studies such as Kiyak *et al*. [14] considered material oxidation as most prevalent on crack flanks. The net effect of this was to reduce the crack driving force (the stress intensity factor range). Therefore, to model this effect on fatigue crack growth behaviour, a time-dependent uniaxial expansion of a layer of finite elements was considered, with a gradual alteration of yield stress. Although the model was able to predict the observed crack growth behaviour [14], the approach could not adequately reflect the mechanism of oxidation.

Crack Growth behaviour.

Fatigue life design and assessment has been a research subject of great interest for over two centuries now. Various theoretical and experimental approaches have been developed to assess life of components. These approaches are considered as design against fatigue and therefore broadly divided into total-life and damage-tolerant approaches. Total-life approaches are mainly applicable to smooth specimens which are theoretically defects free. Damage-tolerant approaches, on the other hand, were based on the understanding that materials are inherently flawed. In total-life predictions, different characteristics of cyclic deformation can be accounted for. These include effects of mean stress, stress concentrations, multiaxial stresses, stress fluctuations due to variable amplitudes and environmental effects. In general, the total-life approach is based on the relationship between stress/strain amplitude and number of cycles to failure (S/N curve). [86–88]. However, considerable deviations from this behaviour are normally reported when different testing environments are considered. A study on the influence testing environment has on the fatigue behaviour of nickel-based superalloys (particularly Inconel 718) has shown that the presence of oxygen leads to significant increases in crack growth behaviour, as compared to tests conducted in vacuum conditions [43, 72, 89].

In gas-turbine engines, nickel-based superalloys are subjected to high static/cyclic loads at high temperatures in non-ambient environments for long periods of time. In addition to high oxidation and corrosion resistance, these materials are expected to possess high strength, fatigue- and creep-damage resistance characteristics. Varying extents of oxidation-accelerated fatigue crack growth behaviour have been reported. This is a direct consequence of a myriad of competitive effects such as extents of oxidation, testing temperature, loading conditions and material inherent microstructural features. Particularly, loading waveforms were found to significantly influence crack growth behaviour. It was reported that, the longer the imposed dwell at peak loads, the higher the growth rates of cracks in both vacuum and air [72, 89]. Additionally, in line with the general behaviour of nickel-based superalloys which show considerable (mechanical properties) temperature dependency [46, 90], crack growth studies at higher temperatures revealed increased growth rates [43, 91].

A combination of the variation of testing temperatures and different durations of imposed dwell at peak loads were also investigated. Results appeared to suggest a further increase in crack growth rates [89]. Influence of grain sizes (resulting from varied heat treatment procedures in polycrystalline superalloys) on the fatigue crack growth behaviour was also investigated. Results showed that coarse grained specimens had lower crack growth rates as compared to fine-grained specimens at all temperatures considered. Imposition of dwells at peak loads did not change the observed behaviour, but only increased the growth rate [1].

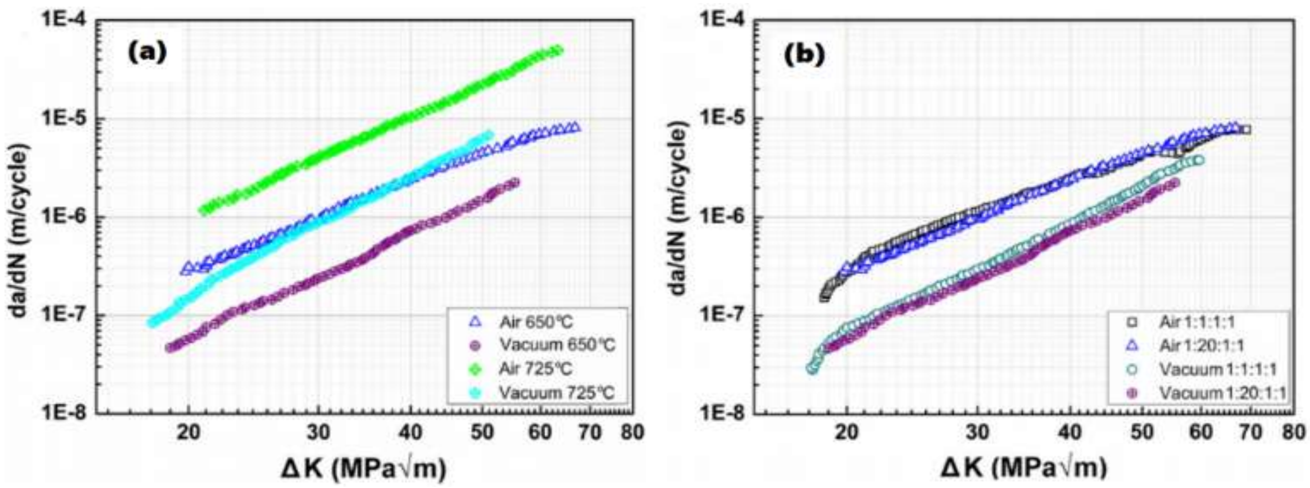

Fig. 7. Influence of testing environment on (a) temperature, and (b) imposition of a dwell at peak load in the alloy Inconel 718 [43].

A study on the influence of oxidation on fatigue crack initiation and propagation in a polycrystalline superalloy appeared to suggest otherwise [43]. Here, although temperature appeared to have similar effects on crack growth behaviour as in the studies by Reed *et al.* [89] and Gustafsson *et al*. [92] (see figure 7a), imposition of a hold time at peak load did not show significant effects. The correlation of the crack growth rate (da/dN) to the stress intensity factor range (ΔK) in figure 7a and b, for tests conducted in air and vacuum. This could be a result of testing temperature. On one hand, Reed *et al.* [89] tested the three superalloys at temperatures between 816ºC and 927ºC. On the other hand, in the work of Jiang *et al.* [43], tests were carried out at temperatures between 650ºC and 725ºC. Additionally, Reed *et al.* [89] considered longer imposed dwells at peak load (2 mins = 120 s) while Jiang *et al.* [43] considered only 20 s.

Overall, it appears that most studies on the influence oxidation has on crack growth behaviour of nickel-based superalloys, concentrated more on polycrystalline alloys. This is evident in the limited availability of published work on directionally solidified and single crystal superalloys. Generally, literature appears to be of the view that, in nickel-based superalloys, imposition of hold times at peak loads, has similar effects on fatigue crack growth behaviour as the increase in testing temperature. This is confirmed by studies conducted on polycrystalline [63, 91, 93], directionally solidified [59, 94] and single crystal [95] superalloys.

CONCLUSIONS

Although the detrimental effects of oxidation damage, on the structural functionality of nickel-based superalloys at high temperatures, were widely reported, it is still not clear how oxidation damage and mechanical loading interact to result in inferior mechanical behaviour. This normally is the case when studies are conducted in oxygen-rich environments as compared to oxygen-deficient environments. Even though oxygen-rich environments were widely cited for the observed enhanced oxidation damage, crack closure effect, reduced creep load bearing capabilities and accelerated crack growth behaviour, to date, only limited work were conducted to provide a clearer understanding of the mechanism by which the interaction between mechanical loading and oxidation damage, lead to inferior mechanical behaviour. As such, studies need to focus particularly on the effect of the interaction between oxidation damage and fatigue loading on crack growth behaviour. Oxidation needs to be considered as a physical process driven by diffusion of oxygen and alloy elements (that form the major phases - *γ* and *γ'* - of nickel-based superalloys, to form oxides such as Cr_2O_3 and Al_2O_3). This will enable establishment of the mechanism by which application of a (cyclic or constant) mechanical load tends to increase oxidation damage, which is still yet to be fully understood. In static oxidation studies, although, kinetics were generally assumed to conform to either linear, parabolic or logarithmic laws, kinetics for individual oxides, such as NiO, Cr_2O_3, Al_2O_3 and spinels – $NiTiO_3$, $NiTaO_4$ and $(Ni/Co)Cr_2O_4$, are still not yet fully understood. This understanding will be necessary in the modelling of the synergetic influence of the interaction between oxidation damage and mechanical loading on the mechanical behaviour of nickel-based superalloys. It is further noteworthy that although the influence of microstructures on oxidation was widely investigated, to the best knowledge of the authors, no study has attempted to established and quantify which one of the two major microstructural features (*γ'-precipitates* or grain boundaries) accounts for more oxidation damage in polycrystalline materials. Furthermore, literature appears to be deficient in knowledge about the optimum sizes of the strengthening phases, the *γ'-precipitates*, and grains necessary for oxidation damage resistance in polycrystalline nickel-based superalloys.

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